Micro-Nano Sensors & Actuators for Use in Extreme Environments

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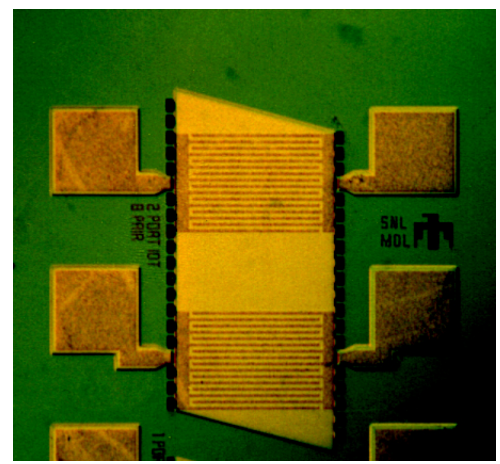
Chad Parish and Paul Galambos
Sandia National Laboratories
John Ekerdt University of Texas at Austin,
Paul Nealey, University of Wisconsin
Jacob Jones, University of Florida

Multilayer Piezoelectric Fuel Injector

NINE Workshop
Marriott Hotel
Albuquerque, NM
July 29, 2008



Bulk Acoustic Surface Wave Piezoelectric Sensor

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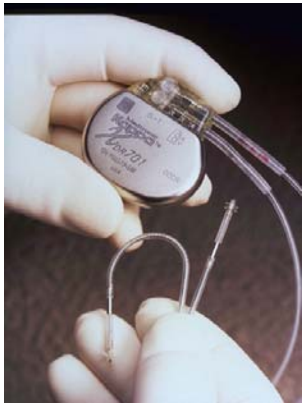
Extreme Environment Sensors and Actuators Needed For Many Applications

Unattended Ground Sensors

Down Hole Well Drilling



Process Control, Integrated Electronics And Bio – Medical

Nano-Micro Materials Advances Required to Improve Sensor Technology

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Example: Pressure Measurement Improvements Needed for Down Hole Well Drilling

There are Many Other Sensor Applications that NINE Teams Can Impact

Improved Sensors Will Save Millions of Dollars for U.S. Down Hole Well Drilling Industry

Improvements Needed

- **Reliably measure small changes in dynamic pressure**
  - **High resolution**
  - **Smaller packages**
  - **Higher Temperature (HT) Performance**
- **Highly Stable Pressure Sensors**
  - **Minimal drift at high temperatures > 150^{o}C**
  - **Replace strain gauges**

Solution: Form NINE Team of Industry, SNL, UTA, Univ of Wisconsin, U of Florida with Materials, Commercial Innovations and Prototype device capabilities to develop microstructure and element patterning at the nanoscale level that will enable next generation HT piezoelectric sensors with improved sensitivity and stability (drift)

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NINE Micro-Nano Sensor Project Will Enable New Well Drill Capabilities Via Materials Advances

Material Advances

Synthesis/Processing
Powder Based
(SNL/UW/UTA CSD/ASD/ PLZT Nano-Synthesis/Patterning)

Electrode – Piezo Interfaces Development
(UTA Ekerdt, J. Jones, UF, SNL)

Materials and Electrical Property Characterization

SNL/Univ/Industry
Prototype Fabrication
(SNL / University
NINE Industrial Partners)

Nanomaterials/Analyses/ Patterning
P. Nealey, UW
J. Ekerdt, UTA
J. Jones, UF; C. Parish SNL

Prototype Fabrication
G. Brennecka, B. Tuttle
P. Galambos (Adv. MEMS)
J. Henfling (Geo Therm Res)

Project Manager:
J. Voigt SNL DM

Principal Investigators:
G. Brennecka,
B. Tuttle SNL Staff

Team Members:
J. Ekerdt (UTA)
P. Nealey (UW)
J. Jones (UF)
J.Henfling, P.Galambos
B.Hernandez, D.Moore
C. Parish (SNL)
Industry

Sandia Has Down Hole Expertise Via Energy Research and Systems Analysis

- Down Hole Life Time Studies SOI microelectronics, Capacitors, Pressure Sensors
- TPS Dewared Memory Tool
- Fluid Sampler
- DWD POC and HT Drilling Tool
- Dewarless PTL monitoring tools (USGS and NETL)
- 300 ^{o}C Analog PT Well Monitoring Tools

200^{o}C SNL Tests of SOI Microelectronics

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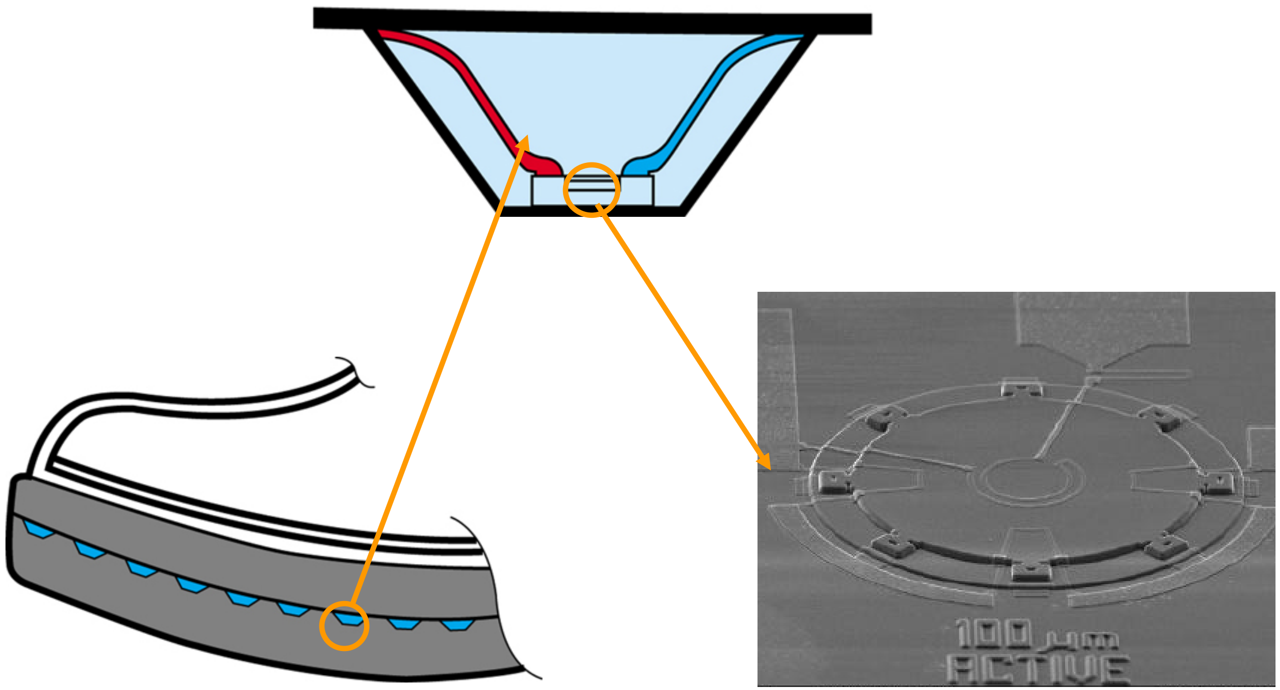
Microelectronic Sensors Developed to Monitor Soldiers for DARPA

- Advanced MEMS Pressure Sensor Design and Fabrication Capability (Paul Galambos)

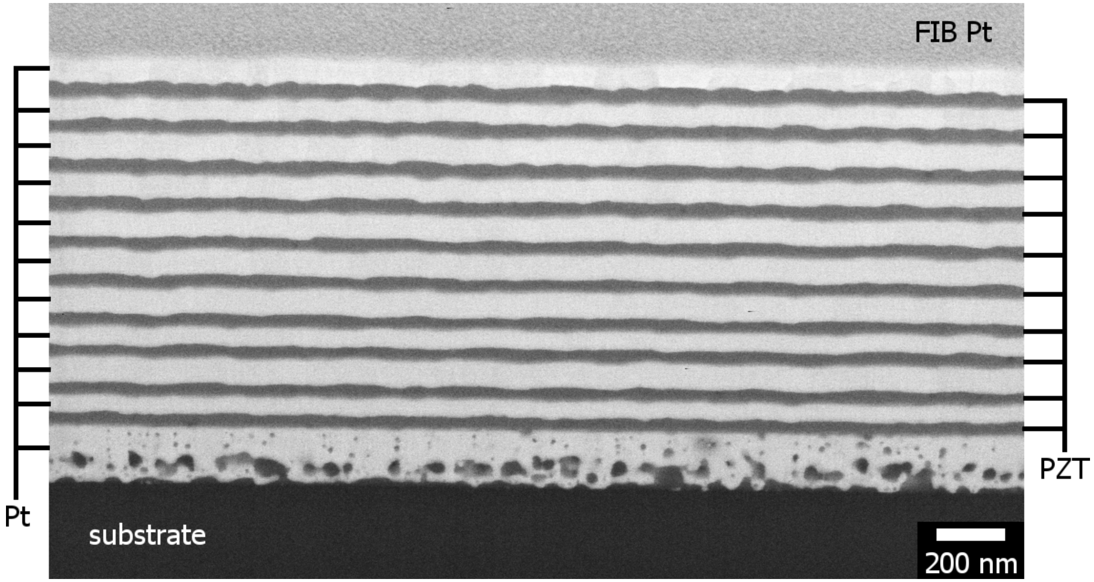


Provides Load History – Monitors soldier activities (DARPA)
Have also Invented a MEMS array Normal Stress Sensor for Monitoring Penetrator Loading

System Needs / Initial Prototypes Drives Materials Advances

- MEMs Devices / PZT based piezoelectrics
  - FPW Devices, Motion Detectors
  - Nanopowder 3D Actuator Elements
  - Bio-Lab on a Chip Applications
  - Ultrathin Film PLZT Multilayer Capacitors
- Metal Contacts
- Non Pb Containing Materials Development

SNL Has Highest Known Large Operating Temperature Range Areal Capacitor Technology



10 layer, 50 nm thick PLZT Layers
1 µF/mm^2

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SNL IMO-based Solution Route

$Zr(OBu)_4 \cdot BuOH + Ti(OiPr)_4$
Blend 5 min

Acetic Acid / Methanol
Chelating agent / Solvent

$Pb(OAc)_4 + La(OAc)_3 \cdot H_2O$
Dissolve at ~90°C

Acetic Acid / Methanol
Adjust Molarity

Spin Coat Deposition
4000rpm/30sec

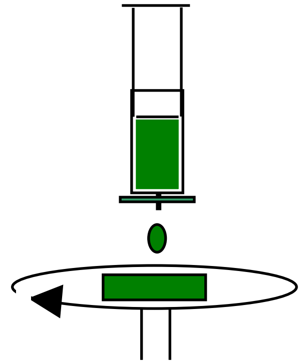
Pyrolysis

Crystallization

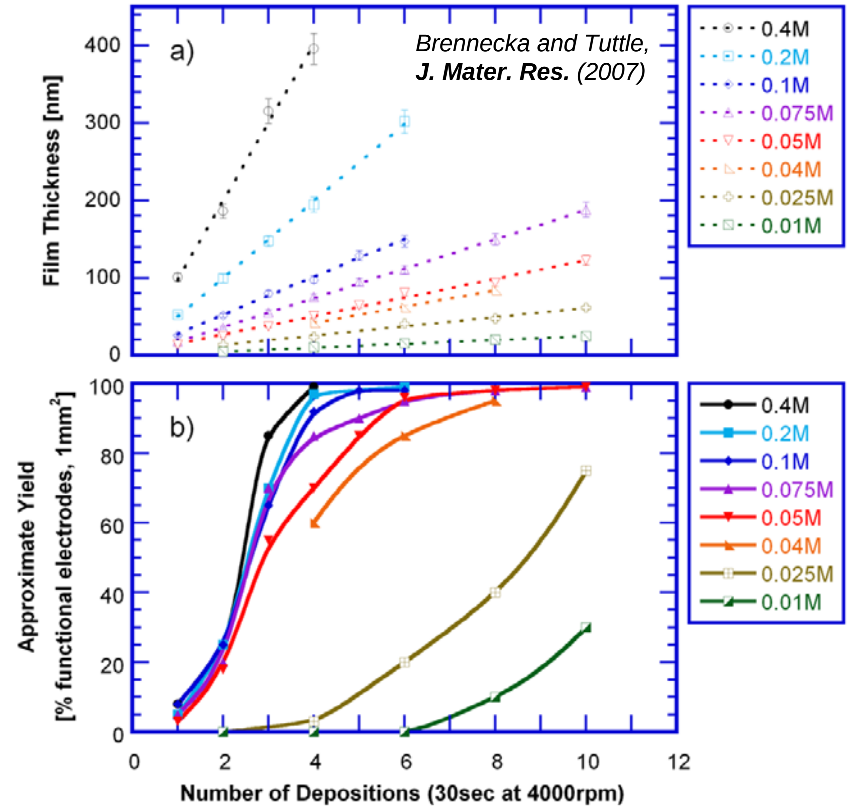
Sputter Pt Electrodes

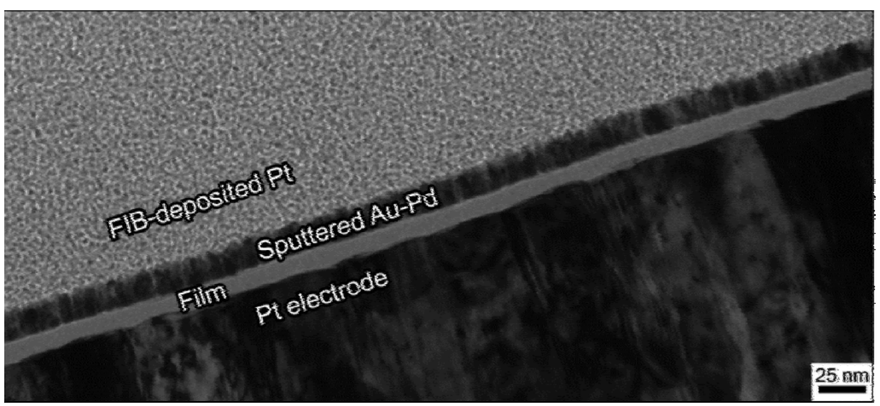
multiple depositions

multiple layers



Spin Deposition





Continuous single-phase films as thin as 9nm

Sigman, Brennecka, Clem and Tuttle.,
***J. Am. Ceram. Soc.** (2008)*

B.A. Tuttle and R.W. Schwartz;
***MRS Bulletin** (1996)*

G. Yi and M. Sayer;
***J. Appl. Phys.** (1988)*

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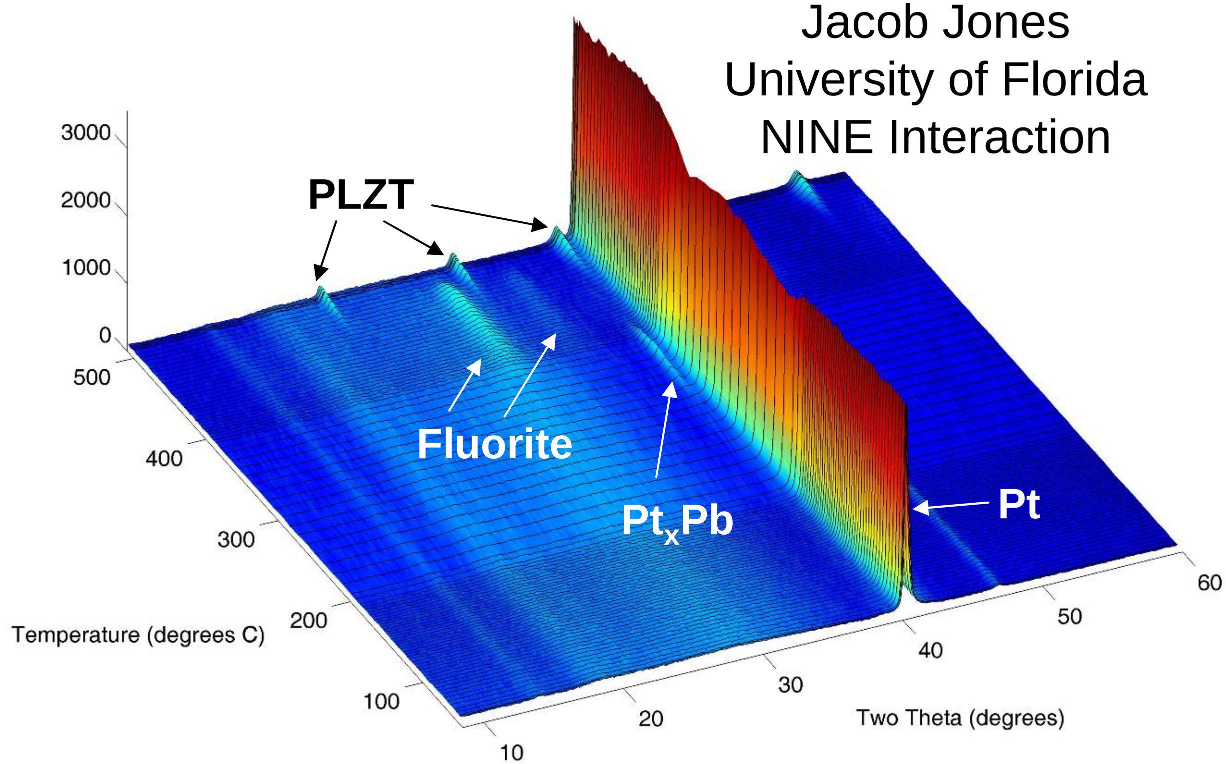
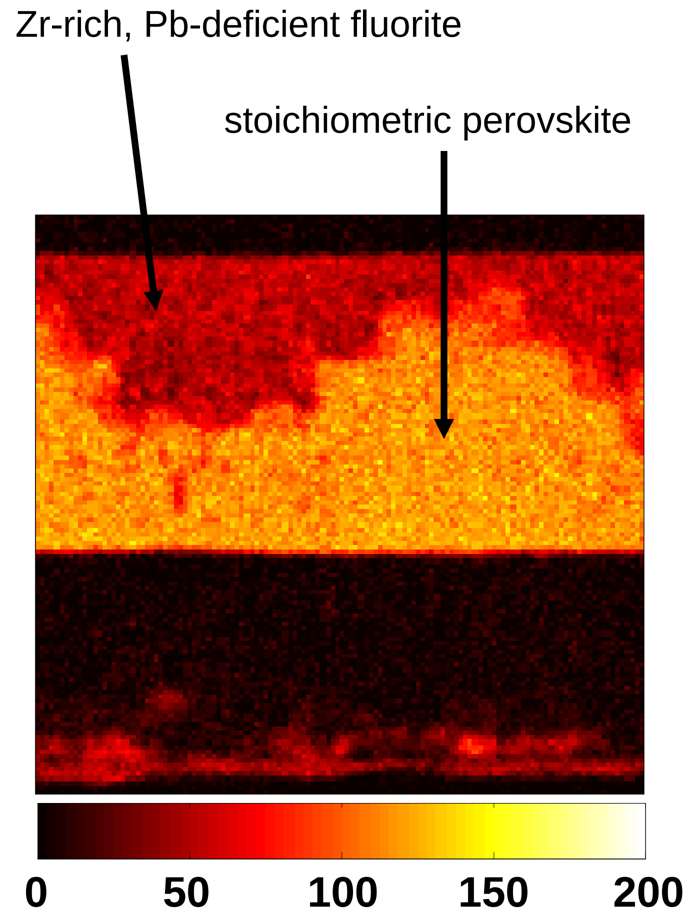
in-situ & *ex-situ* Studies of Phase/Interface Development

Jacob Jones
University of Florida
NINE Interaction





Chad Parish
SNL

- *in-situ* hot-stage XRD reveals presence and effects of transient phases
- *ex-situ* quantitative chemical spectrum imaging in TEM relates chemistry to phase/interface information from XRD with high spatial resolution

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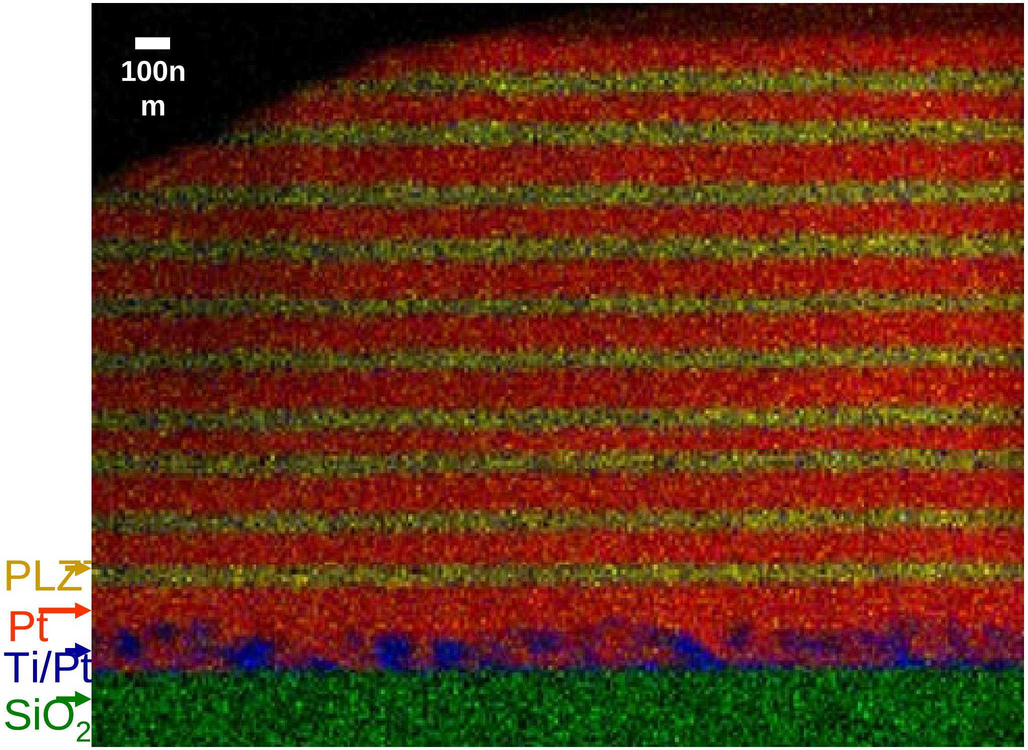
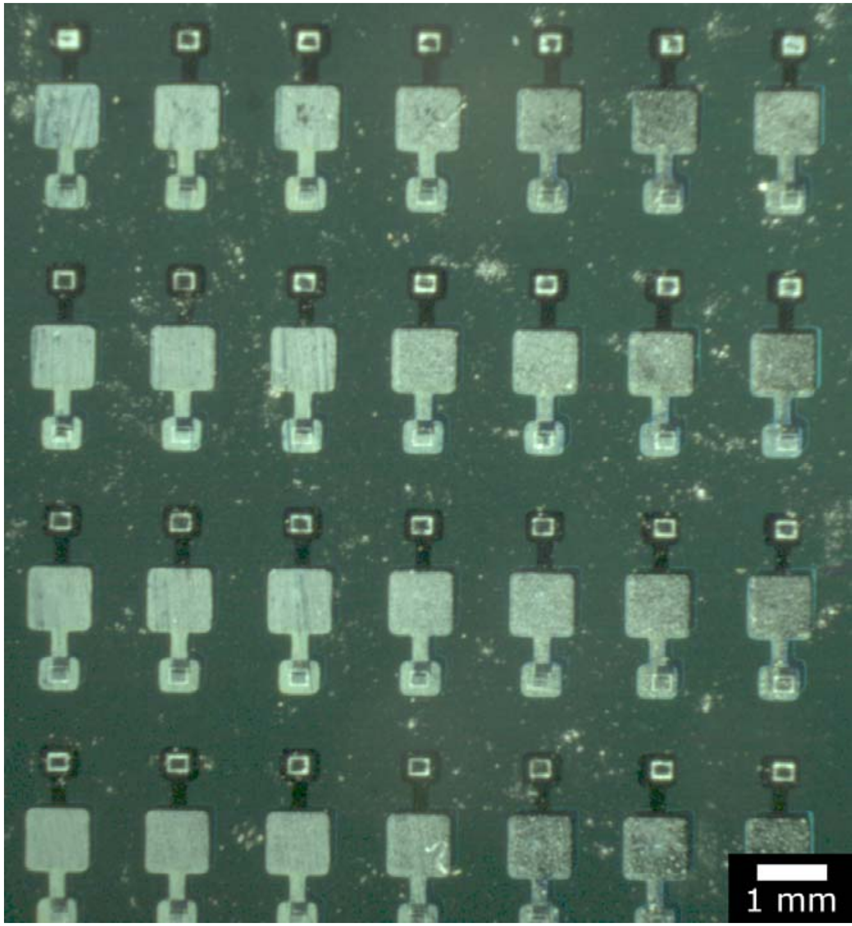
Ten Active Layer PLZT MLC Device, ~50nm

TEM Multivariate Spectral Image of Multilayer Cross-Section



Batch Process Photolithographically Defined PLZT Ultrathin Film Devices



Enhanced Sensitivity per unit volume with thin multilayers

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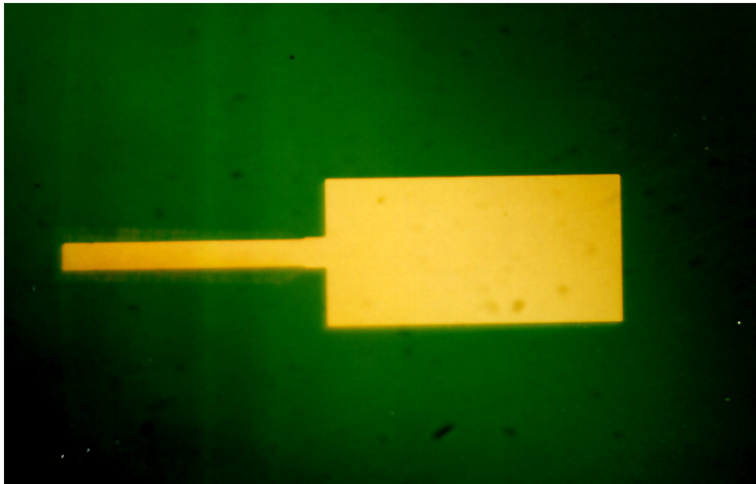
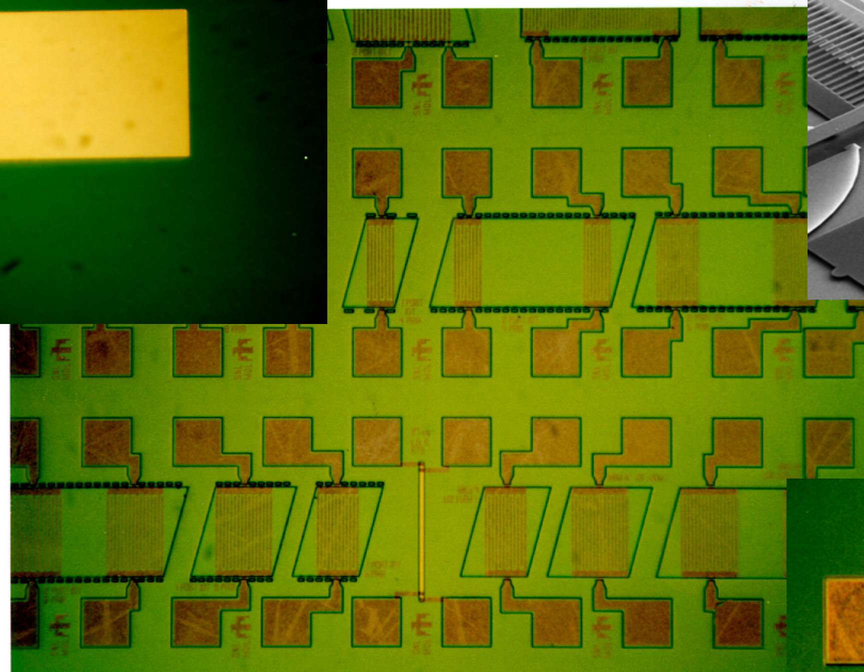
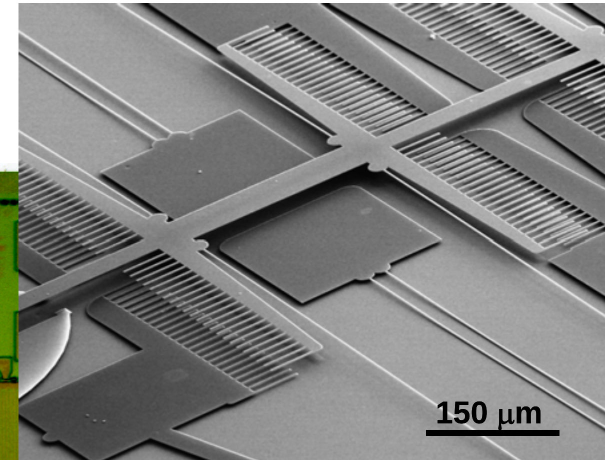
SNL Has Wide Ranging Efforts in MEMS Devices

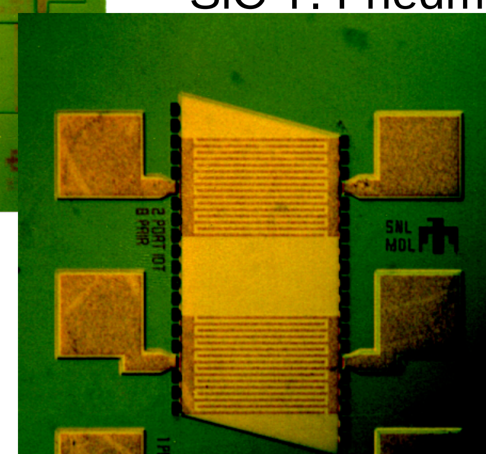




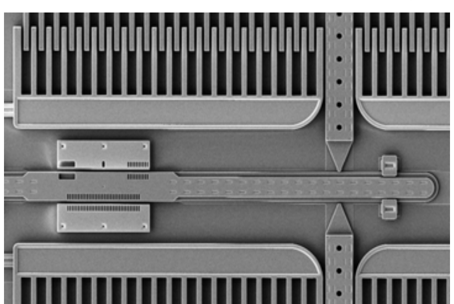


Alternate Hard Materials
ALD To Increase lifetime
SiC T. Friedman

PZT – MEMs
Piezo Cantilever
Beam – Energy
Harvesting



Bulk Acoustic Wave Modulators



Vapor phase lubricants
Extend slide life by 5 orders
Of Magnitude, D. Tanner

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SNL Patented Solution Chemistry Process
Reproducible PZT Ceramics

SNL Chem-Prep Process (Patent) Consists of:

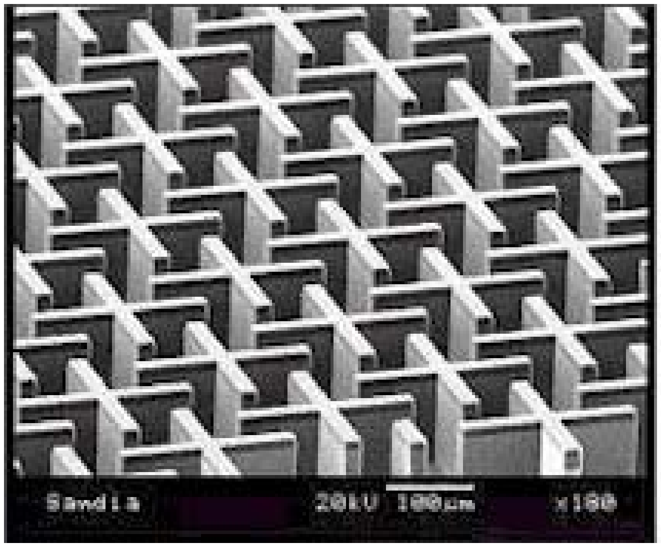
1) Synthesis of Pb Acetate
2) Dissolution of: A) Pb Acetate in Glacial Acetic Acid and
   B) Nb, Zr and Ti n-butoxides in Acetic Acid
3) Blending of the two solutions
4) Precipitation using Oxalic Acid / n-Propanol Solutions

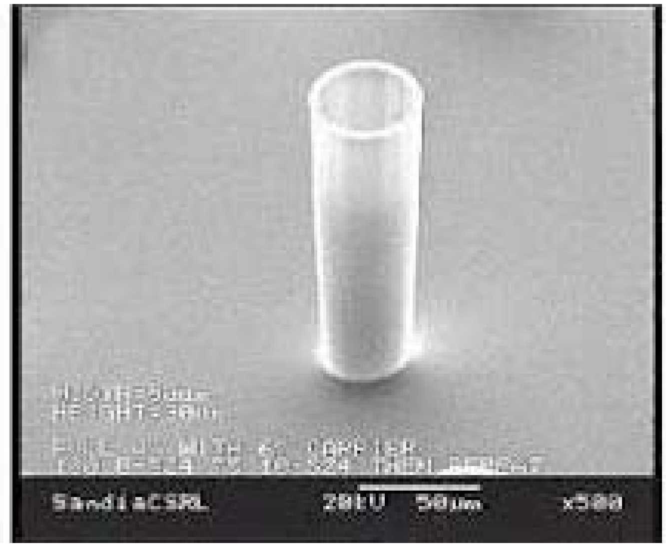
Metal Solution Precipitated
With Oxalic Acid / n-Proponal

More than 100 batches of 1500 grams fabricated,
±0.02 Mol% Ti, ± 0.5% Remanent Polarization; ± 0.1% Density

U.S. Patent No. 5,908,802, "Nonaqueous Solution Synthesis Process for Preparing Oxide Powders of Lead Zirconate Titanate and Related Materials, J. A. Voigt, D.L. Sipola, B.A. Tuttle, and M. T. Anderson,

PZT Nanopowders to Enable Bosch Etched Micromold Patterns in Silicon for Sensors





Micromolding process: size of piezoelectric elements controlled by lithography that can achieve dimensions unachievable using dicing technology.

1) A glass mold of the pattern is prepared using standard lithographic and dry etching techniques.

2) Mold is filled with powder, pressed in standard press and sintered.

3) Excellent materials properties observed in large scale devices

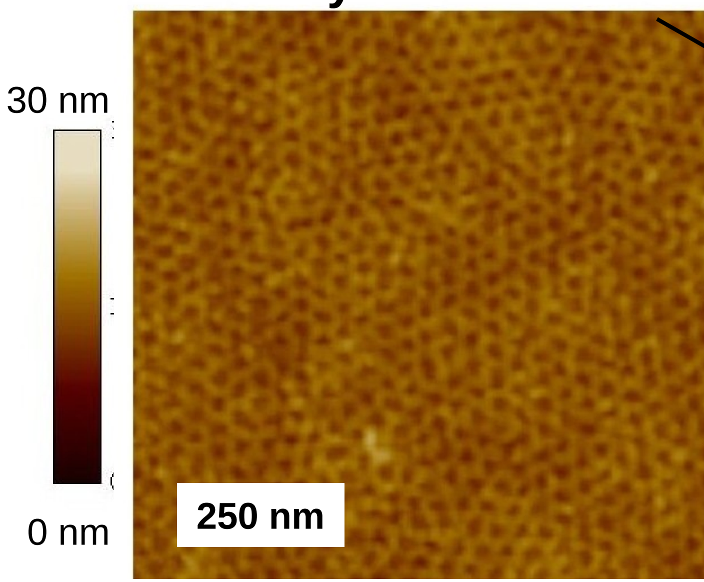
Technical Challenge:

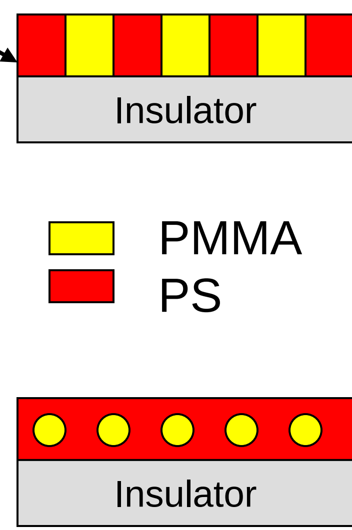
Nanocrystalline, mono-domain grain materials with ultraclean Grain boundaries.

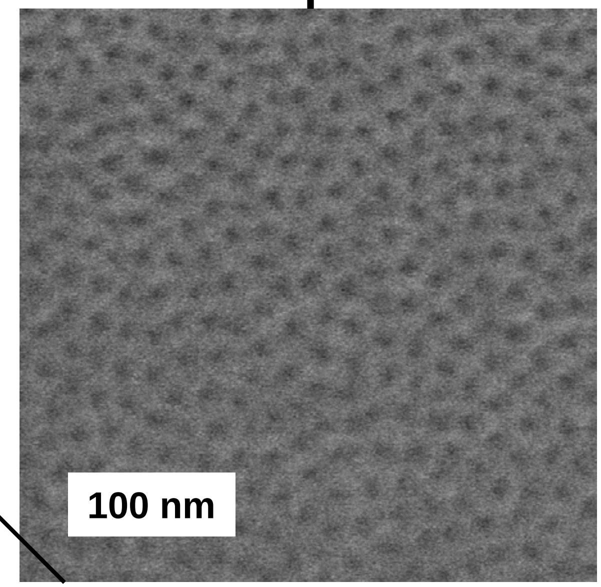
Bilevel Patterning via Graphoepitaxy of Diblock Copolymers (UTA – NINE, J.Ekerdt)

Cylinders

Spheres

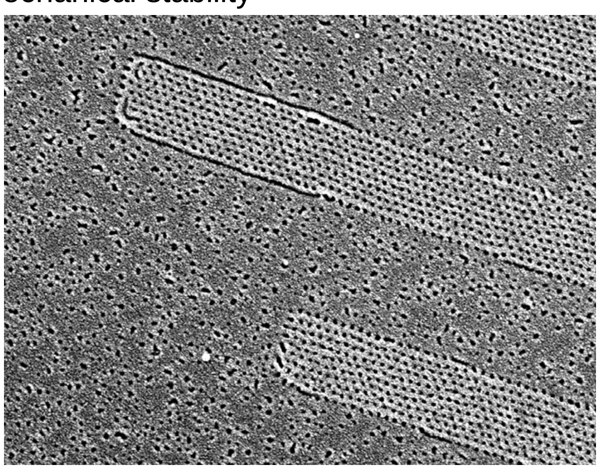






AFM image of cylindrical P(S-b-MMA) template on silicon dioxide after sputtered with gold for mechanical stability

SEM image of spherical P(S-b-MMA) template



<u>PMMA cylinders</u>

70% styrene, 30% methyl methacrylate

20 nm diameter pores, ~6×10^{10} cm^{-2}

<u>PMMA spheres in monolayer</u>

83% styrene, 17% methyl methacrylate

12 nm diameter pores, **~1.2×10^{11} cm^{-2}**

- **If hexagonal, 31 nm center-to-center**

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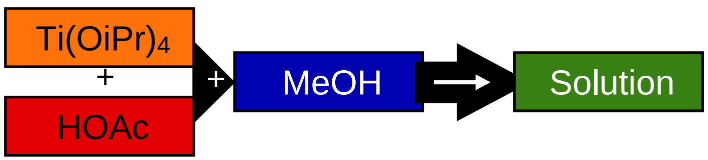
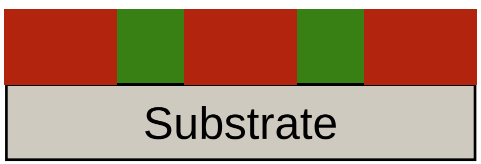
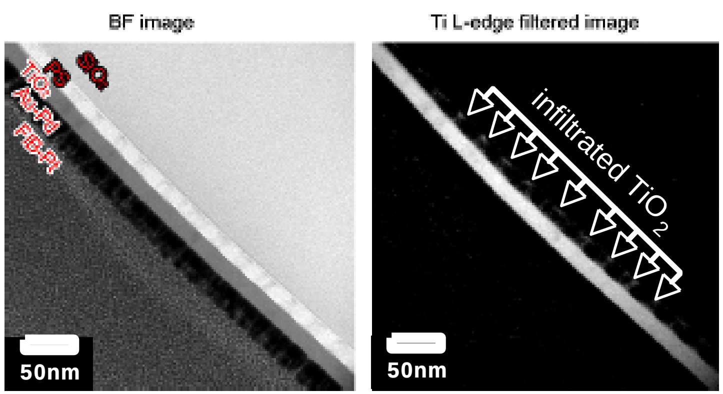
Soft Nanomaterials Development Leads to Patterned Hard Electronic Oxide Nanomaterials

UTA, SNL, UW NINE Collaboration (Paul Nealey UW and Erik Spoerke SNL Soft Matls)

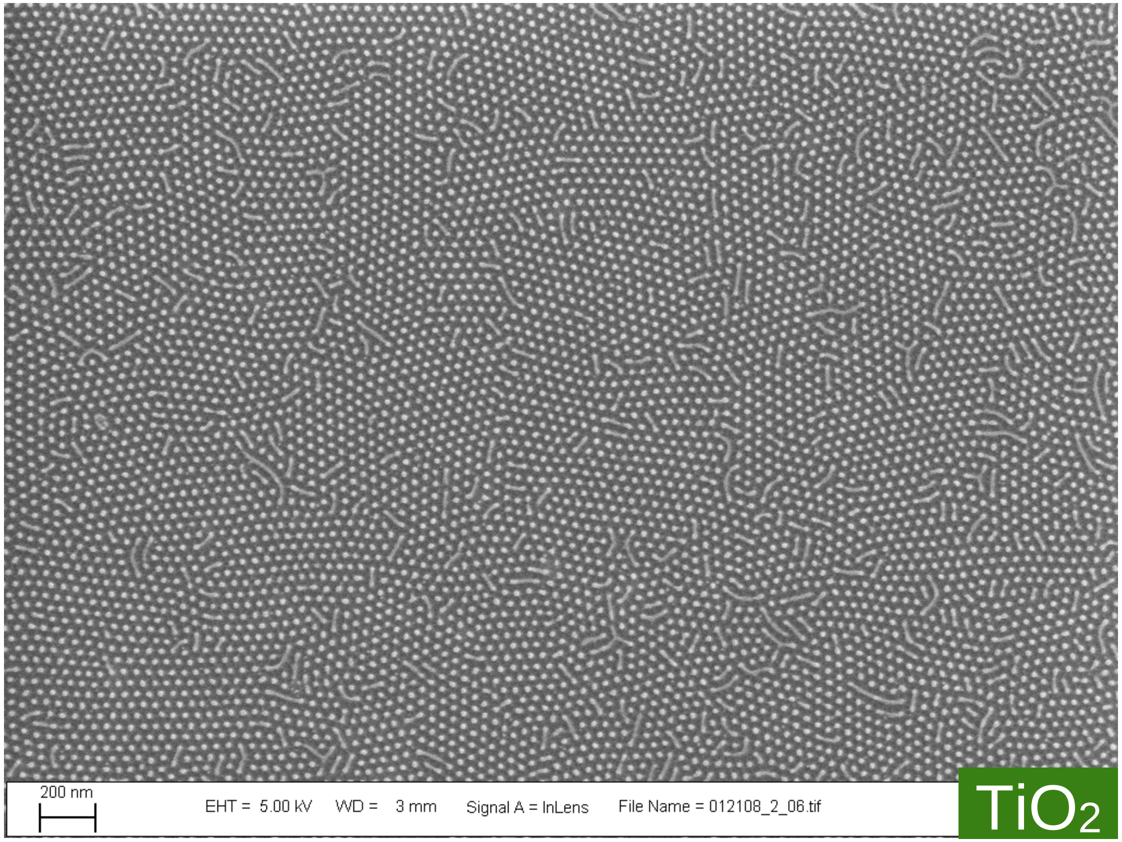


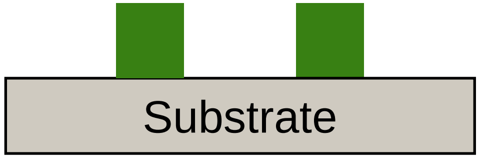


Gel



TiO_2 solution infiltrates 20nm columnar features in nanopatterned PS (also shown for HfO_2)





Remove PS

Also demonstrated with Ni, V, and Mn oxides in nanofeatures; PZT in microfeatures

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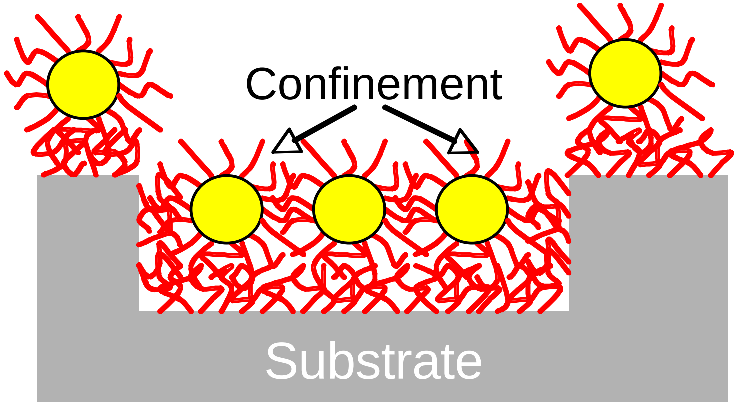
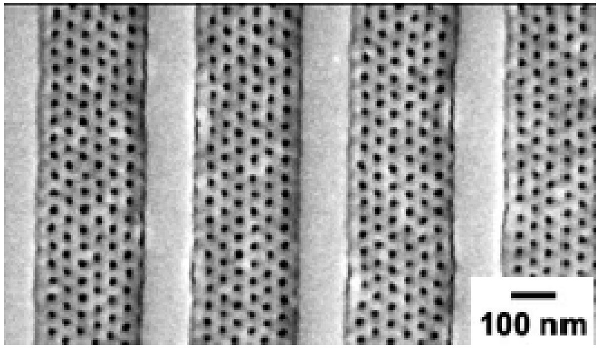
Graphoepitaxy Demonstrated For SiO_2

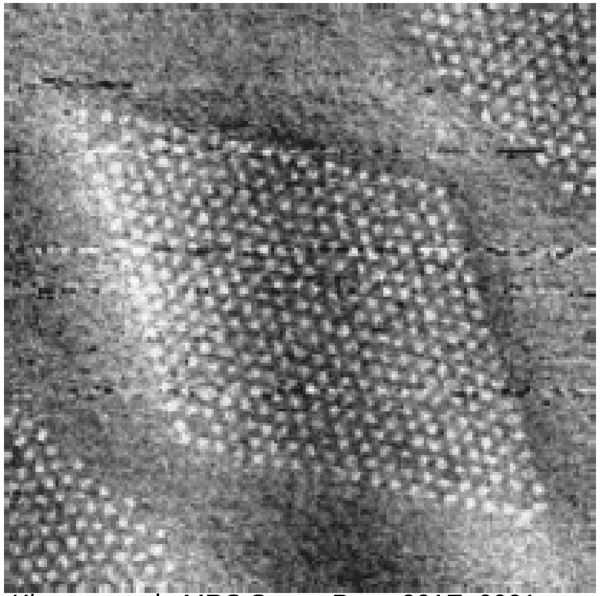
- Geometric confinement can lead to ‘crystalline’ single-domain state
- Demonstrated for Si / SiO_2 with cylindrical and spherical diblocks
- *Pre-defined pixel location*



R.A. Segalman, et al., Macromolecules, 36 4498-4506 (2003)



C. T. Black, et al., IEEE Trans. Nanotechnology 3 (5) 412-415 (2004)

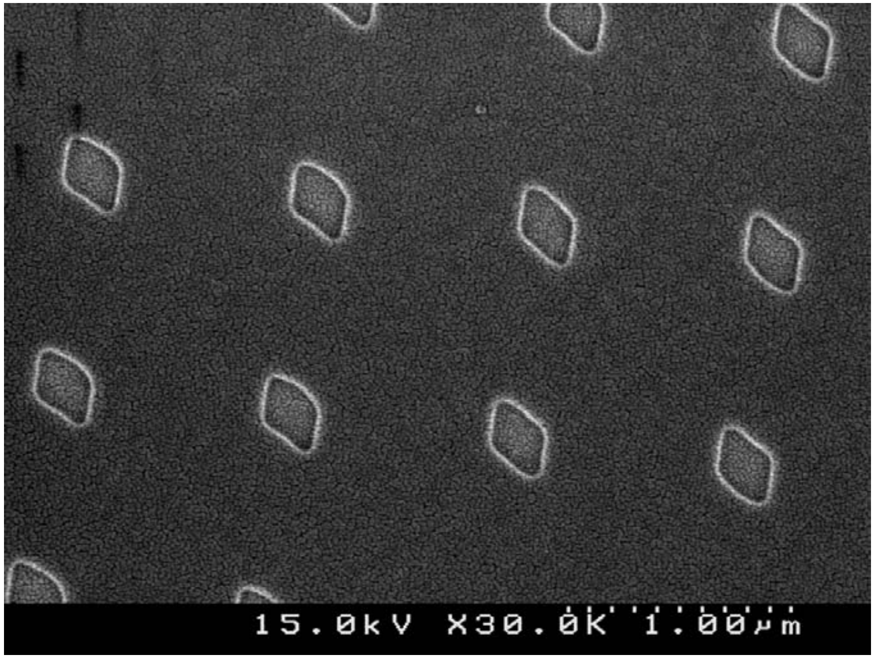


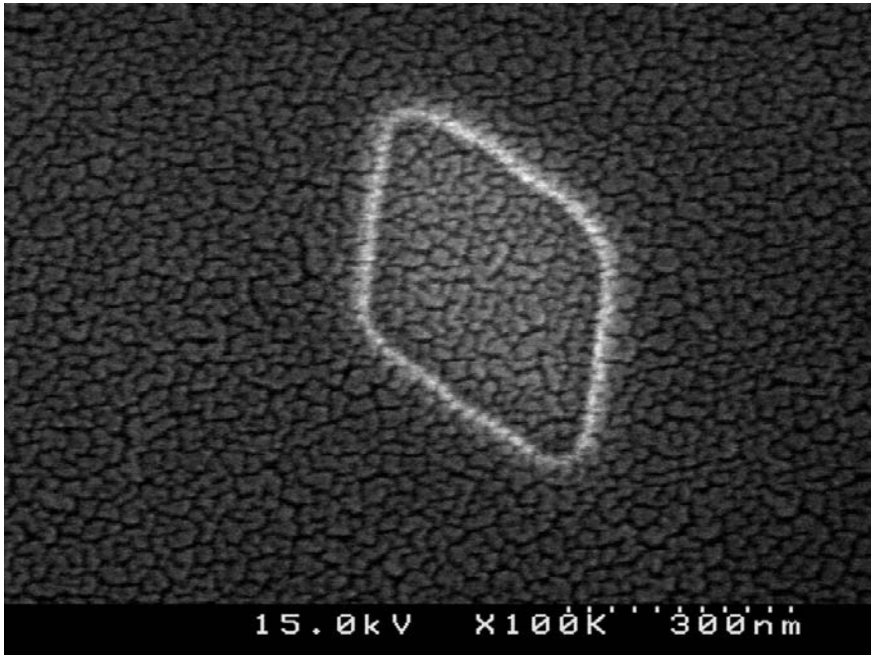
Kimura, et al., MRS Symp. Proc. 901E, 0901-Ra05-05-Rb05-05.1 (2006)

NINE Collaborations Result in Game Changing Technological Advances

- Discussions with (SNL Experts) indicate US industry will Save Millions of Dollars with High Stability, Higher Sensitivity Extreme Environment Sensors
- Wide Range of Applications: Integrated Electronics Monitoring, Process Control, Well Drilling, etc.
- Control of Grain Size of Complex Oxide Materials and Grain Boundary Interfaces on the Nanoscale Essential for Extreme Environment Sensors and Actuators
- Mesh Industry Know How with University Fundamental Nano materials Expertise and SNL Nano-Micro Materials / Device Capabilities Can Result in Substantial Improvements for Extreme Environment Sensors

Transfer etch, SEM results using CINT EBL





~0.3 micron parallelograms, transfer comes from 245 nm template

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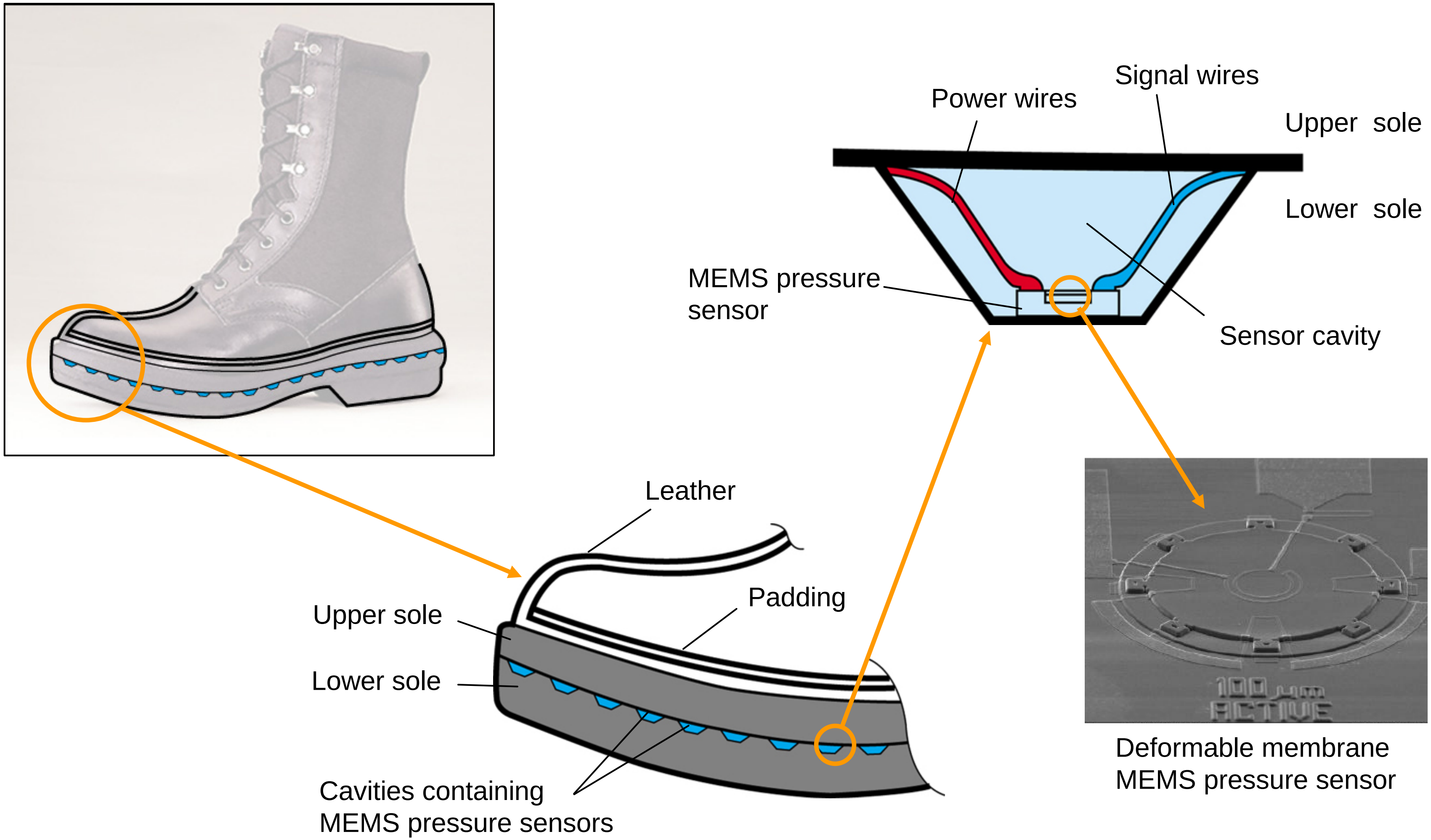
Sandia Developed Tools

- TPS Dewared Memory Tool
- Spectral Gamma Memory Tool
- CTDL Memory Tool
- Fluid Sampler
- Televiewer (Joint with Navy, USGS and Mount Sopris Instrument Co.)
- DWD POC and HT Drilling Tool
- PT Memory Drilling Tools (For Unsen Scientific Drilling Program - Tohoku University)
- Dewarless PT Well Monitoring Tools (For NETL and USGS)
- 300 C Analog PT Well Monitoring Tools

Transducer Packaging (1) ExoBoot: In-sole Pressure Profiler Concept (DARPA)



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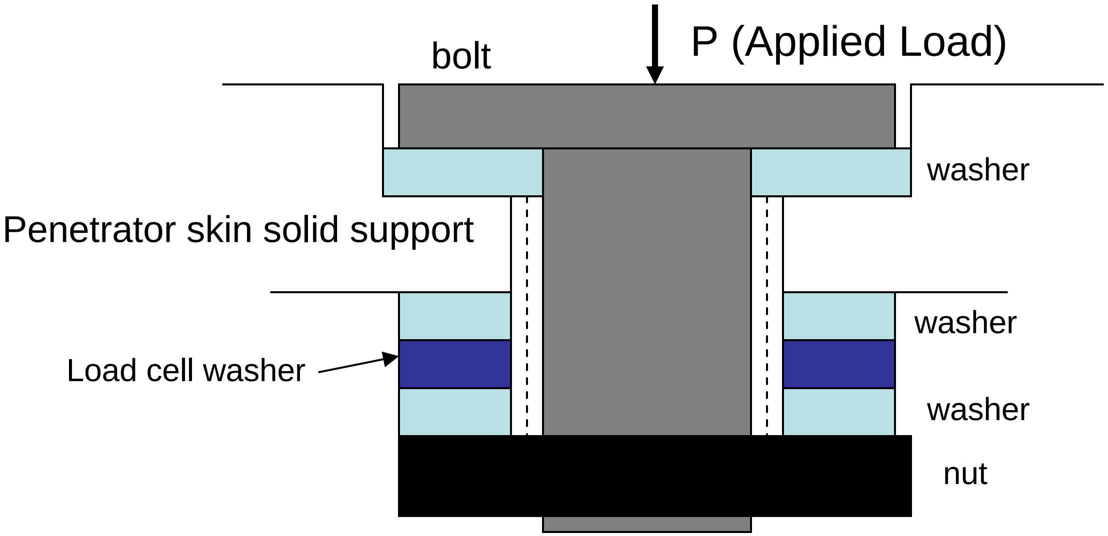
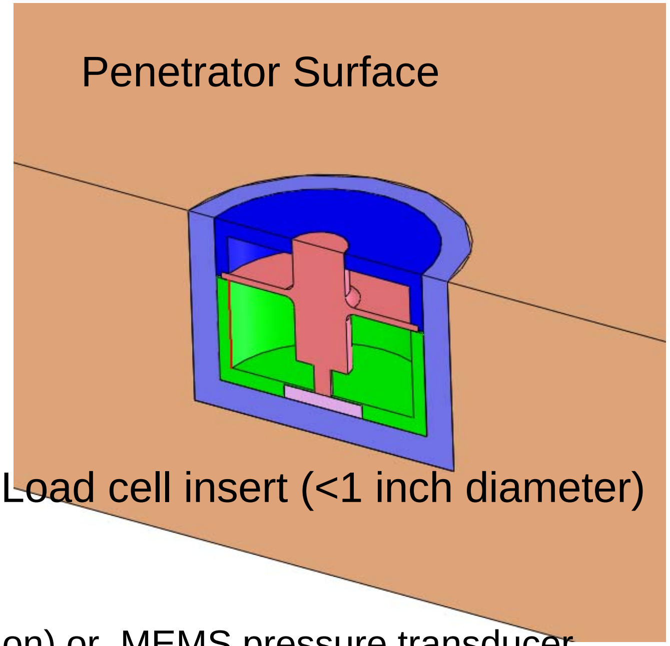
MEMS Normal Stress Sensor for Monitoring Penetrator Loading

Vincent Luk (Department 5431) and Paul Galambos (Department 1769)

Threaded Load Cell Insert in Penetrator Skin.





The load cell washer consists of a commercial load cell (COTS version) or MEMS pressure transducer array (high load MEMS version) and is the sensing element of the Penetrator surface load cell insert.

- COTS version bench test design under way – test this FY.
- MEMS design of pressure sensor array awaiting results of COTS test.
- MEMS pressure sensor array will provide higher load information in a smaller package than COTS load washer. Also, MEMS array will provide a load distribution providing more information about Penetrator surface load details (e.g. load angle, time history…)

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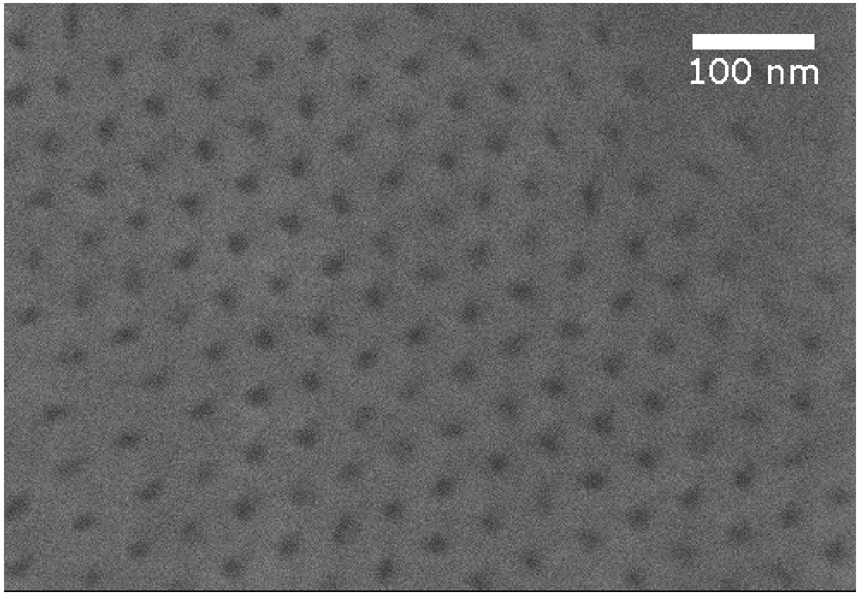
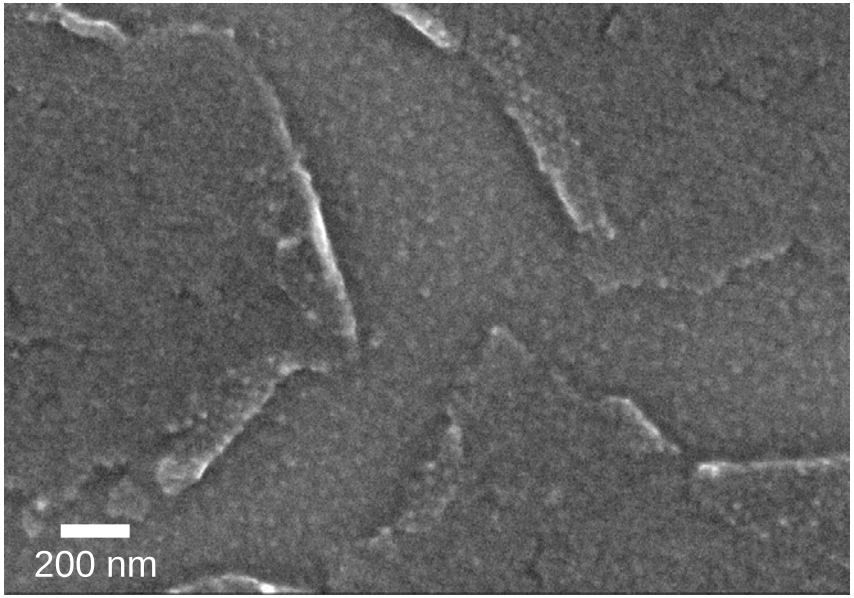
Nanopatterning: TiO_2 deposition

- Very little success since first attempt
- Problems appear to be due to removal of mask layer





- Repeated exact procedure from first trial with longer 'soak' time before spinning; waiting for SEM

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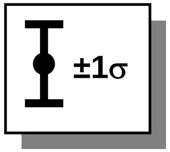
Spectral Image Derived Line Scans: Needed Analytical Tool



Traditional linescans

Extracted from SIs